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Two-Step Sintering of Ceramics

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<http://dx.doi.org/10.5772/68083>

Abstract

Sintering is a critical phase in the production of ceramic bodies. By controlling the density and microstructure formation, sintering now emerged as a processing technology of ceramic materials. Tailoring the structural, mechanical, electrical, magnetic and optical properties is widening the application of ceramics in various fields. Recently, many advanced sintering methods have reported to fabricate ceramic materials with controlled properties. Two-stage sintering (TSS) is one of the simple and cost-effective methods to obtain near-theoretical density materials with controlled grain growth without adding any dopants. Many recent works have reported the use of TSS as a processing method to fabricate nanoceramics for various applications. With this background, this chapter reviews the advantages of TSS in ceramic preparation based on properties and materials and explores the future directions.

Keywords: two-step sintering, grain growth, ceramic properties, densification, sintering mechanism

1. Introduction

Highly dense ceramics with smaller grain size are widely used in high-performance applications in extreme conditions. Sintering is the responsible step for densification of ceramic bodies, and due to its influence on the properties of the material, sintering is also emerging as a new fabrication method. Controlling the powder size [1], use of sintering additives [2], green body density [3–5] and sintering environment [6, 7] and using new sintering methods such as microwave sintering [8, 9], pressure-assisted sintering [10], spark plasma sintering [11] and field-assisted sintering [3] are used for fabrication of dense and fine-grained ceramics. But these may destroy the unique properties of ceramics [12]. Also the applications of new

sintering techniques are limited by low mass productivity, and they are not economically feasible. Two-stage sintering (TSS) is an effective way to achieve fine-grained microstructured ceramics with high densification and relatively low cost. TSS method is successfully applied for all types of ceramics such as structural ceramics, bioceramics, ferrites, piezoelectric ceramics and electrolyte ceramics. Most of the ceramics exhibit controlled or no grain growth in the final stage of sintering and achieved near-theoretical densities. The fine-grained microstructure enhances the mechanical, electrical, magnetic as well as piezoelectric properties of ceramics which widen the applications of ceramics.

TSS consists of heating the samples in two stages. Different sintering profiles were applied in TSS. Generalized diagrams of TSS are shown in **Figure 1(a)** and **(b)**. In the sintering profile 1, the first-step sintering temperature is higher than the second-step sintering temperature, and the first-step holding time is lower than the second-step holding time. Sintering profile 2 is the other way around. The first-step sintering temperature is lower than the second-step sintering temperature, and the first-step holding time is normally higher than the second-step holding time.

In addition, a modified two-step sintering profile is also reported with a cooling step (to room temperature) in between first and second step sintering [2, 13, 14]. Especially, this method used different sintering methods at first and second steps [2, 14].

This chapter mainly focuses on TSS with high first-step sintering temperature. The mechanism of densification with controlled grain growth is explained briefly, and the extended applications of TSS on different ceramics are outlined. Finally the chapter concludes the current trends and challenges of TSS as a fabrication method.

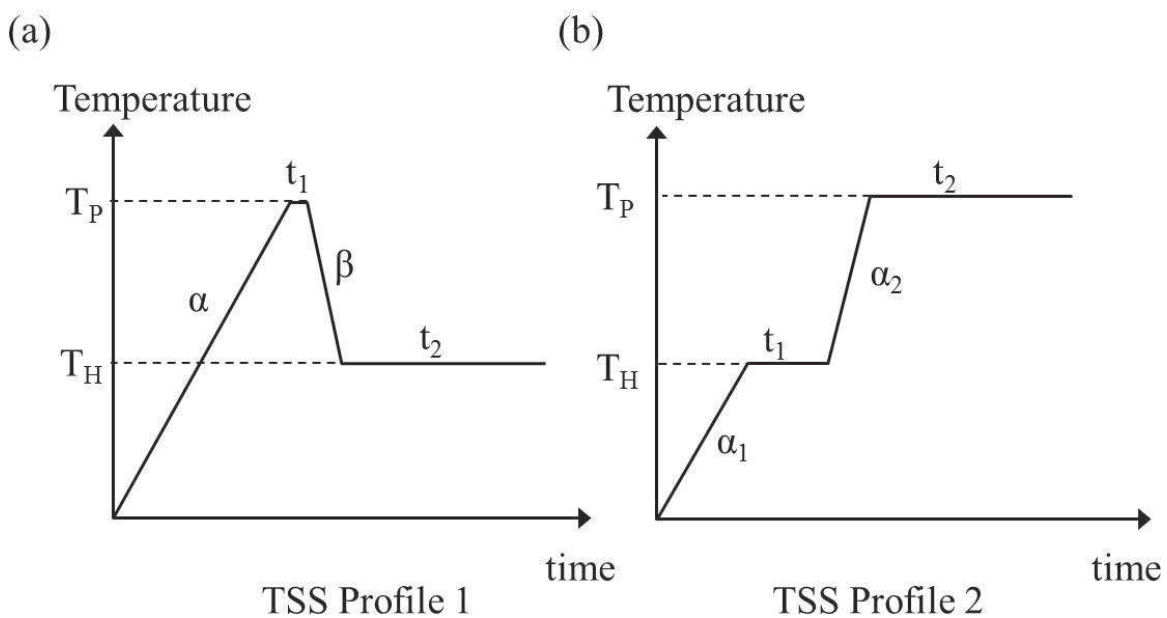


Figure 1. Different sintering profiles used in TSS (a) with high first-step sintering temperature and (b) with a low first-step sintering temperature.

2. TSS with high first-step sintering temperature

TSS with a high first-step sintering temperature is widely used to obtain fully dense ceramics with controlled grain growth due to lower second-step sintering temperature. The sintering profile for TSS with higher first-step sintering temperature is depicted in **Figure 1(a)**. Sintering profile 1 also has few categories based on other sintering parameters:

- Holding time (t_1): The first-step sintering holding time (t_1) is assumed to be zero in few studies, and others hold for a few minutes at temperature T_p .
- Cooling rate (β): Few studies assumed sample rapidly cooled from first sintering temperature (T_p) to second-step sintering temperature (T_H), and others used controlled cooling rates.

The successful TSS method using profile 1 was first introduced for Y_2O_3 by Chen and Wang [15]. In the TSS method, the ceramic samples were heated to a higher temperature to achieve critical density and then immediately cooled to lower temperature and held at that temperature for long holding time to achieve full densification. Density of the sintered sample increased with increasing grain size during the first-step sintering. However, grain growth is controlled at the second stage of sintering, and grain size versus density graph is horizontal at the final stage of the sintering [15]. Similar relationship was observed for other ceramics such as Mg, Nb-doped Y_2O_3 [15], ZnO [16], Ni-Cu-Zn ferrite ceramics, $BaTiO_3$ [17] and $0.89Bi_{0.5}Na_{0.5}TiO_3 \cdot 0.06BaTiO_3 \cdot 0.05K_{0.5}Na_{0.5}NbO_3$ lead free antiferroelectric ceramics [18]. However, the most crucial task in this method is to identify suitable sintering parameters such as heating and cooling rate, the first- and second-step sintering temperatures, holding times and sintering atmospheres.

2.1. Sintering mechanism

TSS with a high first-step sintering temperature is widely used to obtain fully dense ceramics with controlled grain growth due to lower second-step sintering temperature. The mechanism for controlled grain growth in TSS with higher first-step sintering temperature was firstly proposed by Chen and Wang [15], for a TSS study on Y_2O_3 ceramics, and it is widely accepted and verified for other ceramics.

The general mechanisms that are responsible for densification during sintering are grain boundary migration and grain boundary diffusion. Grain boundary migration is responsible for the rapid grain growth in the final stage of conventional sintering. The densification of ceramics with grain growth suppression at the second-step sintering can be explained by the absence of grain boundary migration during the second-step sintering as described in **Figure 2**. In conventional single-step sintering (SSS), the grain growth is accelerated due to grain boundary migration and grain boundary diffusion in the final stage of sintering. Rapid cooling before the second stage of sintering freezes the microstructure by immobilizing the triple-point junctions and continues densification will be achieved by grain boundary diffusion.

For a successful TSS profile with higher first-step sintering temperature, few conditions should be achieved at the end of first-step sintering. The sample must be reaching a critical density at

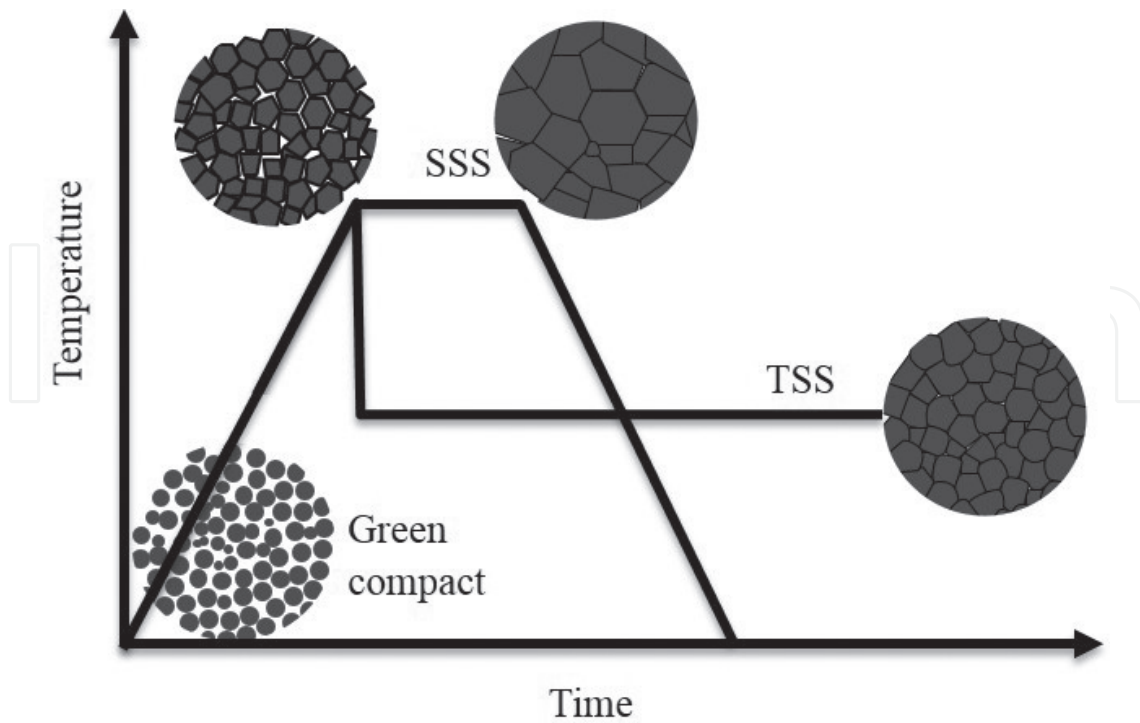


Figure 2. Schematic of densification of ceramics with grain growth during conventional single-step sintering (SSS) and densification without grain growth at lower second-step temperature during TSS with higher first-step temperature.

the end of the first-step sintering so that the densification is active in the final sintering step. This critical density depends on the material. As reported, the critical density to be achieved at the end of the first-step sintering for Y_2O_3 is 75% [15], $BaTiO_3$ is 73% [17] and Ni-Cu-Zn ferrite ceramic is 76% [17]. The critical density is essential to ensure that the pores in the material are subcritical and unstable against shrinkage which can be filled by grain boundary diffusion in the second-step sintering.

A kinetic window will be used to identify the optimum range of the first-step sintering temperature for successful TSS. **Figure 3** illustrates the kinetic window for pure and Mg- and Nb-doped Y_2O_3 . The filled squares between the lower and upper limits of the first-step sintering represent the successful second-step sintering. The open squares above the upper limit of the first-step sintering show grain growth during the second step and below the lower limit represents the failed attempts to achieve full densification. It can also be concluded that the kinetic window can be shifted up and down along the first-step temperature axis with the addition of dopants. So far, the kinetic windows have been proposed to Y_2O_3 [15, 19], Mg, Nb-doped Y_2O_3 [15, 19], ZnO [20], Ni-Cu-Zn ferrite [17], $BaTiO_3$ [17] and $(1-x) BiScO_{3-x} PbTiO_3$ (BSPT) [21].

Another important study on exploring the mechanism of grain growth suppression in TSS with higher first-step sintering temperature was reported on ZnO ceramics. The transmission electron microscope (TEM) images of a TSS sample at a first-step sintering temperature of 800°C for 60 s and a second-step sintering temperature of 750°C for 20 h (**Figure 4(a)**) and a conventionally sintered ZnO at 1100°C (**Figure 4(b)**) were examined for the evidences of grain boundary migration. Ten different grain boundary zones were analysed from the TEM

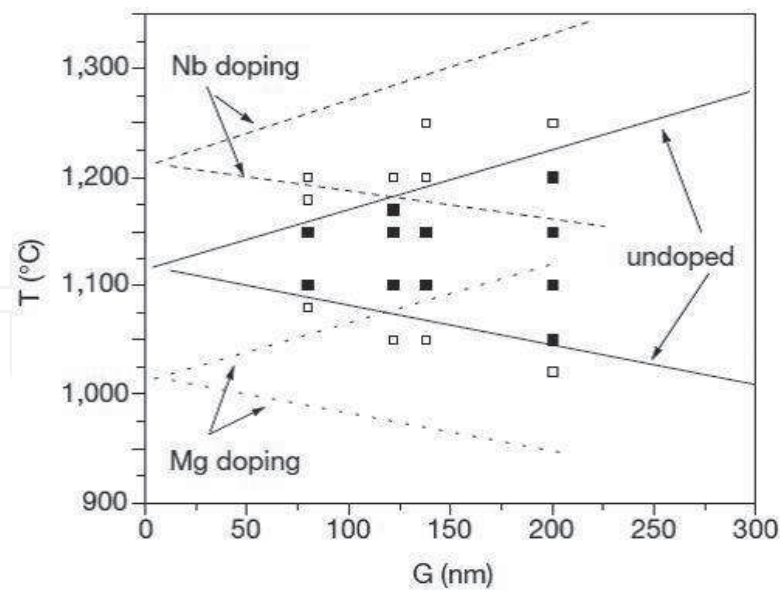


Figure 3. Kinetic window for pure, Mg- and Nb-doped Y_2O_3 (T is the first-step sintering temperature and G is grain size) [15].

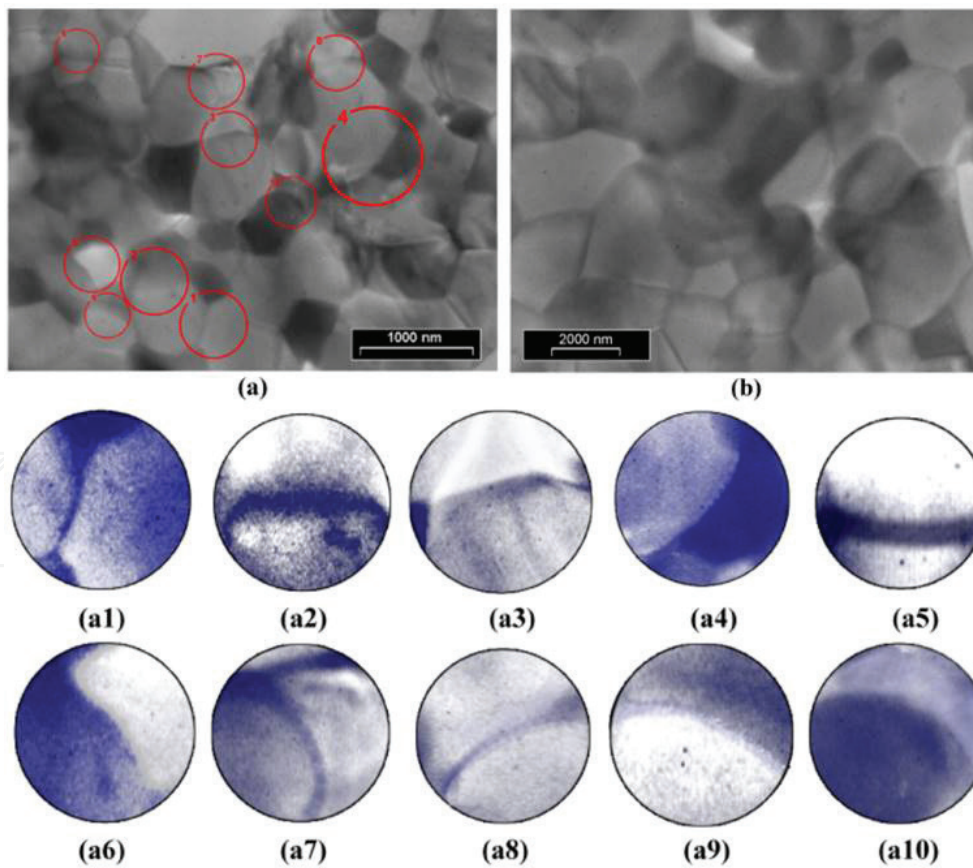


Figure 4. TEM images of the ZnO samples sintered (a) using TSS profile with the first-step sintering temperature of 800°C for 60 s and a second-step sintering temperature of 750°C for 72,000 s, (b) conventionally at 1100°C and (a1)–(a10) magnified images of grain boundaries marked in circles at (a) [22].

image of TSSZnO sample. As can be seen in **Figure 4(a1–a10)**, junctions seem to have pinned the boundaries of growing grains, and the curvature of these boundaries resulted from the mentioned immobilized triple points. However, no similar zones are observed in the TEM images from conventionally sintered samples (**Figure 4(b)**).

2.2. Effect of TSS with high first-step temperature on properties of ceramics

By successfully controlling the grain growth, the TSS with higher first-step sintering temperature is used to fabricate many ceramic materials with enhanced properties that are used for advanced applications [23]. So far TSS is applied for 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) [1, 4, 5, 8, 24–31], 8 mol% yttria-stabilized zirconia (8YSZ) [3, 5, 9, 24, 32–34], Al_2O_3 [5, 24, 25, 35–43], doped alumina [37, 41], yttrium aluminium garnet (YAG) [44, 45], magnesium aluminium silicate [46], corundum [42, 43], hydroxyapatite [14, 47–50], forsterite (Mg_2SiO_4) [51], TiO_2 [11, 52–54], SrTiO_3 [25] ($\text{K}_x\text{Na}_{1-x}$) NbO_3 (KNN) ceramic [12, 55–57], SiC [58–60] and Si_3N_4 [2] and used for several applications. This section outlined the changes in properties of various ceramics sintered using different TSS profiles.

2.2.1. Sintering of zirconia ceramics

Pure zirconia has three crystallographic structures, monoclinic, tetragonal and cubic. At room temperature monoclinic is stable. In pure form, zirconia has very low appeal for use as engineering ceramic due to reverse transformation on cooling resulting in severe cracking associated with volume expansion ($\sim 3\%$) to the monoclinic phase. In order to overcome this problem, stabilizers such as MgO, CaO and Y_2O_3 are added. Stabilizers reduce the change of chemical free energy and stabilize tetragonal or cubic phase at room temperature.

2.2.1.1. 3Y-TZP ceramics

3Y-TZP is also known as ceramic steel, which exhibits excellent mechanical properties. It is found in many applications, such as cutting tools, grinding media for powders, extrusion dies and biomedical application. Fully dense ceramics with uniform microstructure and fine grain size is essential to stabilize the tetragonal phase as well as to improve the mechanical properties such as hardness and toughness. There are many TSS profiles conducted to control the grain growth of Y-TZP ceramics [1, 4, 5, 8, 24–31, 61]. Only few researchers successfully obtained fully dense ceramics with controlled grain growth (< 5 times of powder size) [1, 4, 5, 25, 28, 29, 31, 62]. Among these studies, Binner et al. [31] and others [1, 29] successfully sintered nanostructured zirconia ceramics. Binner et al. [31] achieved fully dense (99% of TD) nanoceramics with the application of hybrid radiant and microwave sintering. However, the second-step grain growth was not entirely suppressed in both radiant and microwave sintering, but the rapid growth observed in conventionally sintered samples was controlled. Application of TSS also enhances the hardness (12–14 GPa), fracture toughness 5–9 $\text{MPa m}^{1/2}$ and bending strength (900–1100) MPa [61, 62].

Despite the fulfilment of zirconia in a wide variety of application, it suffers from low-temperature degradation in the humid atmosphere around 65–500°C, with hinder biomedical application. Recently Sutharsini et al. [62] sintered fully dense (99% of TD) 3Y-TZP via TSS with average grain size 290 nm. The sintered ceramics exhibited better hydrothermal ageing resistance against the conventionally sintered 3Y-TZP ceramics.

2.2.1.2. Sintering of 8YSZ

8YSZ ceramic is a promising candidate as a solid electrolyte for fuel cell application. Maca et al. [5] reported that efficiency of TSS depends on the crystal structure of the ceramics. The authors claimed that cubic structure has higher efficiency in controlling growth than hexagonal and tetragonal structures. Hence, TSS on 8YSZ controls the grain growth compared to 3Y-TZP due to its cubic crystal structure. TSS has been widely applied to 8YSZ to improve mechanical, electrical as well as the gas permeance via controlling the grain growth [3, 9, 24, 32–34]. It enhance the hardness (~ 13 GPa) [9, 32, 63], fracture toughness ($3 \text{ MPam}^{1/2}$) [9, 32, 63] and ionic conductivity (0.3 Scm^{-1}) [9]. In addition to the above property, it is necessary to control grain growth to achieve optimum ratio of grain size to electrolyte layer thickness ($0.1 < z < 0.3$) to control gas permeance value for SOFC [64]. TSS effectively controls gas permeance value [26].

Hesabi et al. [9] reported that the TSS is efficient in obtaining fine grain 8YSZ when compared to conventional and microwave sintering with nearly full density. The grain size and conductivity of conventionally sintered sample at 1500°C with a heating rate of 5°C/min, microwave-sintered samples at 1500°C with a heating rate of 50°C/min and TSS with the first-step sintering temperature of 1250°C with no holding time and the second-step sintering temperature of 1050°C with 20 h holding time were compared. The grain sizes were reported as 2.15 μm , 0.9 μm and 295 nm, and the conductivity at 1000°C were 255.4, 322.6 and 398.6 mS/cm for conventional, microwave and two-step sintered samples, respectively.

2.2.2. Sintering of aluminium-based ceramics

Alumina-based ceramics are widely used for optical and biomedical applications. It is also used as filler for plastic and cutting inserts. However, the brittle nature of alumina limits the application. Ultrafine grain microstructure is crucial to enhance the mechanical properties such as hardness, wear resistance and strength. TSS successfully applied doped and pure alumina [5, 24, 25, 35–41]. Bodišová et al. [36] and others [5, 25, 35, 37, 39] successfully sintered fully dense alumina (98%) with controlled grain growth (grain growth below five times than that of powder size). Furthermore, the sample sintered with TSS exhibits excellent hardness (18 GPa) and fracture toughness ($4 \text{ MPam}^{1/2}$) [35].

It is also reported that the room temperature cooling between the first and second steps of sintering also affects the grain growth suppression of corundum abrasives. The TSS with 1250°C first-step temperature and 1350°C with a second-step temperature of 1150°C for 5 h yielded fine-grained corundum abrasives with grain sizes of 65 and 80 nm, respectively. The samples sintered at the same sintering profiles with a room temperature cooling yielded grain sizes of 400 and 560 nm, respectively. The conventional sintering at 1300°C for 2 h resulted to a final grain

size of 800 nm [43]. In addition, the two-step sintered corundum abrasive exhibited excellent hardness (22 GPa), fracture toughness ($3 \text{ MPam}^{1/2}$) and wear resistance ($<2 \times 10^{-7} \text{ mm}^3/\text{Nm}$) [43].

2.2.3. Sintering of YAG ($\text{Y}_3\text{Al}_5\text{O}_{12}$)

YAG is a familiar ceramic material for luminescent materials. Presently, single crystalline YAG is applied in lasers pumped by solid-state LEDs, scintillators, and infrared windows. Generally, YAG is sintered for high temperature and long holding time which leads to abnormal grain growth. TSS is an efficient and economic method, which control the abnormal grain growth and improved the transmittance of YAG [44, 45]. YAG sintered with the first-step sintering temperature at 1800°C and the second-step sintering temperature $1550\text{--}1600^\circ\text{C}$ revealed more than 40% transmission [44, 45]. Furthermore, neodymium-doped YAG (Nd:YAG) sintered via TSS exhibited excellent transmittance (85%) [65].

2.2.4. Sintering of hydroxyapatite (HA)

Hydroxyapatite is a bioceramic that is used as tissue implants due to its excellent biocompatibility. However, low toughness hinders application of artificial bone and teeth implants. Furthermore, the major drawback of HA is that it decomposed into secondary phases (α - or β -tricalcium phosphate). In order to avoid such decomposition, TSS has been applied to HA [14, 47–51, 66, 67]. Feng et al. [14] and others [14, 49, 50, 66, 67] successfully sintered monophase HA without decomposition. Furthermore, TSS improved mechanical properties. Mazaheri et al. [50] achieved highest hardness (7.8 GPa) and fracture toughness ($1.9 \text{ MPam}^{1/2}$) via TSS.

2.2.5. Sintering of Ni-Cu-Zn ferrite

Ni-Cu-Zn ferrite ceramics received special attention due to its low cost, excellent heat and corrosion resistance, high magnetic permeability and low magnetic loss. It is used in many electronic devices such as multilayer capacitor, sensors, antennas and broadband transformers. The electromagnetic properties of Ni-Cu-Zn ferrite are controlled by its microstructure and densification. Wang et al. [17] and Su et al. [68, 69] successfully sintered Ni-Cu-Zn ferrite by using TSS. Wang et al. [17] proposed kinetic window for successful TSS. Ni-Cu-Zn ferrite sintered by using TSS exhibited excellent magnetic properties [68, 69]. Magneto-dielectric materials with matched permeability and permittivity are promising candidates as loading materials to reduce the physical dimensions of low-frequency antennas. Ni-Cu-Zn ferrite sintered via TSS revealed almost equal permeability and permittivity of around 11.8. And the magnetic and dielectric loss tangents were lower than 0.015 in a frequency range from 10 to 100 MHz. These properties make the material useful to the design of miniaturized antennas [69].

2.2.6. Sintering of Si-based ceramics

Silicon carbide is widely used for abrasives and refractories due to its high strength, hardness and excellent thermal shock resistance. In conventional single-step sintering, abnormal grain growth is progressed due to its high sintering temperature. Generally, the grain

growth during the sintering in the presence of liquid phase is much more significant than that of solid-state sintering. Therefore, it is practically impossible to obtain nanostructured ceramics by conventional single-step liquid-phase sintering. TSS was successfully applied in liquid-phase-sintered SiC ceramics, and a fully dense nanostructured SiC ceramic with a grain size of ~40 nm has been obtained [58, 59] in argon atmosphere. Magnani et al. [60, 70] also successfully sintered doped SiC via TSS with enhanced mechanical properties. The sintered samples exhibited excellent hardness (24 GPa), fracture toughness (3 MPam^{1/2}), Young modulus (400 GPa) and flexural strength (500 MPa).

Similar to SiC, silicon nitride, also non-oxide ceramics, exhibits high hardness strength and thermal shock resistance. It is widely applied to automotive engine wear parts due to its outstanding mechanical properties and wear resistance. Bimodal microstructure of silicon nitride was successfully sintered by using TSS without using β -Si₃N₄ seed crystal [10, 71]. Bimodal microstructure enhances strength and toughness of the ceramic via crack bridging toughness mechanism [72]. Barium aluminosilicate-reinforced silicon nitride sintered via TSS also exhibited higher flexural strength (565 MPa) and fracture toughness (7 MPam^{1/2}). The obtained composite exhibits excellent mechanical properties compared to unreinforced barium aluminosilicate matrix [10].

The forsterite (Mg₂SiO₄) ceramic is a new bioceramic with good biocompatibility. Forsterite sintered with the first-step sintering temperature 1300°C and the second-step sintering temperature at 750°C for 15 h revealed high density (98.5%) with grain size 300 nm. Furthermore it exhibited fracture toughness of 3.61 MPam^{1/2}. Compared with hydroxyapatite ceramics, forsterite shows a significant improvement in the fracture toughness. Authors suggested that the two-step sintering method can be used to fabricate improved forsterite dense ceramics with desired bioactivity and mechanical properties that might be suitable for hard tissue repair and biomedical applications [51].

2.2.7. Sintering of alkaline niobate-based lead-free piezoelectric ceramics

Environmental friendly lead-free alkaline-based niobate ceramics exhibited excellent piezoelectric properties compared to Pb(Zr,Ti)O₃ ceramics. Alkaline-based niobate (KNN) ceramics are successfully sintered by using TSS, and they exhibited excellent dielectric [12, 55, 73], ferroelectric [12, 55, 56, 73] and piezoelectric properties [12, 21, 55, 56]. Furthermore (K_{0.4425}Na_{0.52}Li_{0.0375})(Nb_{0.8925}Sb_{0.07}Ta_{0.0375})O₃ exhibited excellent temperature stability over a wide range of temperature, which is attractive for piezoelectric applications [12].

2.2.8. Sintering of zinc oxide

Zinc oxide has been widely applied to electronic and optical devices. Furthermore, alumina-doped ZnO is used as an alternative to indium-doped tin oxide (ITO) as a transparent conductive electrode in photovoltaic devices and displays. Electrical and optical properties of ZnO are mainly influenced by grain size. Grain growth of ZnO was successfully controlled using TSS [16, 20, 22, 74–78]. Zhang et al. [20] and others [16] successfully sintered fully dense

ZnO without grain growth at the final stage of sintering. Furthermore Zhang et al. proposed kinetic window for successful TSS profile. Mazaheri et al. [22] confirmed the triple-point drag mechanism for controlled grain growth at the second-step sintering proposed by Chen and Wang [15] by using TEM image of two-step sintered ZnO compacts. ZnO sintered via TSS exhibited excellent I–V characteristics [16, 74, 76]. ZnO varistors sintered via TSS exhibited higher breakdown field of 6–8 kVmm⁻¹ and nonlinear coefficient of over 270 due to fine grain size and high concentration of ZnO–ZnO grain contacts [16].

2.2.9. Sintering of BaTiO₃

Barium titanate (BaTiO₃) is a polycrystalline piezoelectric ceramic. It is widely used to piezoelectric transducers, sensors and actuators. Many TSS studies have been conducted on BaTiO₃ [17, 79–90]. Barium titanate ceramic is widely applied to multilayered ceramic capacitors (MLCC), transducers and pyroelectric detectors due to its dielectric, ferroelectric and piezoelectric properties. Wang et al. [17, 83] successfully sintered fully dense nanostructured ceramic and proposed kinetic window for successful TSS. TSS not only improved the densification and grain growth but also enhanced dielectric and piezoelectric properties [79, 81, 82, 85–88, 91, 92]. TSS samples exhibited excellent piezoelectric constant 519 pN/C and relative permittivity of 6079 [82, 92]. Tian et al. [86] reported that TSS revealed excellent dielectric constant of 2400 at room temperature, low dielectric loss (<1%) and high insulation resistivity of 10¹² Ωcm, which could be beneficial for multilayer capacitor application.

3. TSS with low first-step sintering temperature

In few TSS studies, samples were initially heated to lower temperatures and then to higher temperature as shown in **Figure 1(b)**. Here the first step is normally a pre-coarsening step that is performed for several purposes including removal of volatile fraction and smoothing the pore channels. This method was successful to prepare fully dense nano-sized pure ZrO₂, fully dense (>98%) alumina [38, 40] and alumina-doped zirconia (7.5Al₂O₃–92.5ZrO₂) (vol.%) [8].

Sintering of pure zirconia has major drawback due to its reversible tetragonal-to-monoclinic phase transformation associated with shape deformation. Tartaj and Tartaj [93] applied TSS for pure zirconia below the phase transition temperature (<1150°C). In the first step, the compact allowed to achieve 96% of the density at 950°C for 10 h, and then the second-stage sintering temperature increased to 1050°C and achieved fully dense crack-free pure zirconia with grain size less than 200 nm.

Fully dense (99%) alumina-doped zirconia (7.5Al₂O₃–92.5ZrO₂) (vol.%) is also successfully sintered by using microwave TSS by using lower first-step sintering temperature. Furthermore, microwave-assisted TSS revealed higher density (99%), hardness (13 GPa), fracture toughness (12 MPam^{1/2}) and bending strength (750 MPa) than the conventional single-step sintering. Alumina-toughened zirconia is widely applied to dental implant due to its excellent biocompatibility and hardness [8].

Al₂O₃ ceramics are also known as a better translucent with gas-impermeable properties which is suitable for high-pressure lamp envelopes. The high optical transmittance requires special efforts to eliminate any light scattering centres such as residual pores, grain boundaries, secondary phases and rough surfaces in the material. MgO-doped alumina ceramic sintered by TSS with lower first-step sintering exhibited improved the transmittance compared to conventionally sintered sample [40].

4. Conclusion

Despite long holding times, TSS with higher first-step sintering temperature is convenient to achieve fully dense and fine-grained microstructured ceramics with improved properties. The TSS is successfully applied to a range of ceramic materials, and their application is broadened. TSS also helped the emergence of sintering as a fabrication technique. Tailoring the TSS conditions and theoretical studies on TSS mechanisms will make TSS a cost-effective method to fabricate advanced ceramics. TSS can also be studied by using different sintering methods and sintering environments in the first and second steps of sintering.

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